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PTO/SB/05 (4/98)
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UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 C.F.R. § 1.53(b))

Attorney Docket No.	Mo-5666/HR-231
First Inventor or Application Identifier	Birgit Schleifenbaum
Title	Encapsulated Flavorings
Express Mail Label No.	EF080092312US

JC966 U.S. PTO 11/06/00

JC948 U.S. PTO 09/707240 11/06/00

APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility patent application contents.

ADDRESS TO: Assistant Commissioner for Patents
Box Patent Application
Washington, DC 20231

1. [X] * Fee Transmittal Form *(e.g., PTO/SB/17)*
(Submit an original and a duplicate for fee processing)

2. [X] Specification [Total Pages 14]
(preferred arrangement set forth below)
- Descriptive title of the Invention
- Cross References to Related Applications
- Statement Regarding Fed sponsored R & D
- Reference to Microfiche Appendix
- Background of the Invention
- Brief Summary of the Invention
- Brief Description of the Drawings *(if filed)*
- Detailed Description
- Claim(s)
- Abstract of the Disclosure

3. [ ] Drawing(s) *(35 U.S C. 113)* [Total Sheets]

4. Oath or Declaration [Total Pages 2]
a. [X] Newly executed (original or copy)
b. [ ] Copy from a prior application (37 C.F.R. § 1.63(d))
(for continuation/divisional with Box 16 completed)
i. [ ] DELETION OF INVENTOR(S)
Signed statement attached deleting inventor(s) named in the prior application, see 37 C.F.R. §§ 1.63(d)(2) and 1.33(b).

** NOTE FOR ITEMS 1 & 13 IN ORDER TO BE ENTITLED TO PAY SMALL ENTITY FEES, A SMALL ENTITY STATEMENT IS REQUIRED (37 C.F.R. § 1.27), EXCEPT IF ONE FILED IN A PRIOR APPLICATION IS RELIED UPON (37 C.F.R. § 1.28).*

5. [ ] Microfiche Computer Program *(Appendix)*

6. Nucleotide and/or Amino Acid Sequence Submission
(if applicable, all necessary)
a. [ ] Computer Readable Copy
b. [ ] Paper Copy (identical to computer copy)
c. [ ] Statement verifying identity of above copies

ACCOMPANYING APPLICATION PARTS

7. [X] Assignment Papers (cover sheet & document(s))
8. [ ] 37 C.F.R.§3.73(b) Statement *(when there is an assignee)* [ ] Power of Attorney
9. [ ] English Translation Document *(if applicable)*
10. [X] Information Disclosure Statement (IDS)/PTO-1449 [X] Copies of IDS Citations
11. [ ] Preliminary Amendment
12. [X] Return Receipt Postcard (MPEP 503)
(Should be specifically itemized)
13. [ ] * Small Entity Statement(s) *(PTO/SB/09-12)* [ ] Statement filed in prior application Status still proper and desired
14. [X] Certified Copy of Priority Document(s)
(if foreign priority is claimed)
15. [ ] Other: .....

16. **If a CONTINUING APPLICATION,** *check appropriate box, and supply the requisite information below and in a preliminary amendment:*
[ ] Continuation [ ] Divisional [ ] Continuation-in-part (CIP) of prior application No _____/_____
Prior application information: Examiner _____ *Group / Art Unit:* _____
For CONTINUATION or DIVISIONAL APPS only: The entire disclosure of the prior application, from which an oath or declaration is supplied under Box 4b, is considered a part of the disclosure of the accompanying continuation or divisional application and is hereby incorporated by reference. The incorporation can only be relied upon when a portion has been inadvertently omitted from the submitted application parts.

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Name (Print/Type)	Noland J. Cheung	Registration No. (Attorney/Agent)	39,138
Signature		Date	11/06/2000

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See 37 C F R §§ 1 27 and 1 28

TOTAL AMOUNT OF PAYMENT ($)910.00

Complete if Known	
Application Number	To Be Assigned
Filing Date	Herewith
First Named Inventor	Birgit Schleifenbaum
Examiner Name	
Group / Art Unit	
Attorney Docket No.	Mo-5666/HR-231

METHOD OF PAYMENT (check one)

1. [X] The Commissioner is hereby authorized to charge indicated fees and credit any overpayments to

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Deposit Account Name: Bayer Corporation

[X] Charge Any Additional Fee Required Under 37 CFR §§ 1 16 and 1 17

2. [ ] **Payment Enclosed:**
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FEE CALCULATION

1. BASIC FILING FEE

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description	Fee Paid
101	690	201	345	Utility filing fee	710.00
106	310	206	155	Design filing fee	
107	480	207	240	Plant filing fee	
108	690	208	345	Reissue filing fee	
114	150	214	75	Provisional filing fee	

SUBTOTAL (1) ($) 710.00

2. EXTRA CLAIM FEES

	Extra Claims		Fee from below	Fee Paid
Total Claims 13	-20** = 0	x	18	= 0
Independent Claims 5	- 3** = 2	x	80	= 160
Multiple Dependent				= 0

**or number previously paid, if greater, For Reissues, see below

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description
103	18	203	9	Claims in excess of 20
102	78	202	39	Independent claims in excess of 3
104	260	204	130	Multiple dependent claim, if not paid
109	78	209	39	** Reissue independent claims over original patent
110	18	210	9	** Reissue claims in excess of 20 and over original patent

SUBTOTAL (2) ($) 160.00

FEE CALCULATION (continued)

3. ADDITIONAL FEES

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description	Fee Paid
105	130	205	65	Surcharge - late filing fee or oath	
127	50	227	25	Surcharge - late provisional filing fee or cover sheet.	
139	130	139	130	Non-English specification	
147	2,520	147	2,520	For filing a request for reexamination	
112	920*	112	920*	Requesting publication of SIR prior to Examiner action	
113	1,840*	113	1,840*	Requesting publication of SIR after Examiner action	
115	110	215	55	Extension for reply within first month	
116	380	216	190	Extension for reply within second month	
117	870	217	435	Extension for reply within third month	
118	1,360	218	680	Extension for reply within fourth month	
128	1,850	228	925	Extension for reply within fifth month	
119	300	219	150	Notice of Appeal	
120	300	220	150	Filing a brief in support of an appeal	
121	260	221	130	Request for oral hearing	
138	1,510	138	1,510	Petition to institute a public use proceeding	
140	110	240	55	Petition to revive - unavoidable	
141	1,210	241	605	Petition to revive - unintentional	
142	1,210	242	605	Utility issue fee (or reissue)	
143	430	243	215	Design issue fee	
144	580	244	290	Plant issue fee	
122	130	122	130	Petitions to the Commissioner	
123	50	123	50	Petitions related to provisional applications	
126	240	126	240	Submission of Information Disclosure Stmt	
581	40	581	40	Recording each patent assignment per property (times number of properties)	40.00
146	690	246	345	Filing a submission after final rejection (37 CFR § 1.129(a))	
149	690	249	345	For each additional invention to be examined (37 CFR § 1.129(b))	

Other fee (specify) _____

Other fee (specify) _____

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SUBTOTAL (3) ($) 40.00

SUBMITTED BY

				Complete (if applicable)	
Name (Print/Type)	Noland J. Cheung	Registration No (Attorney/Agent)	39,138	Telephone	777-2827 (412)
Signature				Date	11/06/2000

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ENCAPSULATED FLAVORINGS

FIELD OF THE INVENTION

The invention relates to a method of producing flavoring-containing particles as well as flavoring-containing particles whose surface has been treated with an inert gas. The resultant encapsulated flavorings are used for flavoring food products and pharmaceuticals.

BACKGROUND OF THE INVENTION

Encapsulated flavorings having a particularly long shelf life are produced in the flavoring industry generally by emulsifying the flavoring in molten carbohydrate mixtures with subsequent shaping. Within an extruder for example, an emulsified flavoring is added to the carbohydrate melt and is then extruded through a perforated plate into a previously charged cooled isopropanol bath. US Patents 4,707,367; 4,499,112; 3,704,137; 3,041,180; 2,809,895 describe processes of encapsulating flavorings that use such a solvent bath. In the isopropanol bath, the strands are comminuted during the solidification by an agitator to particle sizes between about 0.3 and 1.5 mm. This solvent bath serves to wash the flavoring contents, which adhere to the particle surface of the resultant particles. The encapsulated flavorings are then dried to remove the remaining solvent by centrifugation and gentle vacuum drying.

The sugar residues must be removed by fine filtration from the solvent used for washing. Water and flavoring residues are recovered by distillation, for recycling. Despite these downstream operations, contaminated solvents must be temporarily stored in separate tanks. The entire production process must comply with a high safety standard. The plant must be made explosion-proof and the flexibility with respect to product change is very restricted because of the problems of contamination.

Disadvantages of these solvent bath processes are due to the complex solvent treatment. Additionally problematic, the resulting particles have a low glass transition temperature and therefore readily form lumps.

By using an extruder and solidification in air, shaped strands can also be produced without using a solvent. This procedure is disclosed in US Patents 5,603,971; 5,601,865; 5,087,461; 5,786,017; 5,009,900; 4,820,534 and European Patents WO 94/06308; WO 94/23593. Shaped strands are cooled via a cooling belt or the like. The comminution must be carried out in a downstream mechanical comminution process such as a pelletizer or crusher. During the comminution to the desired particle size between about 0.3 and 1.5 mm, the surface structure of the particles is damaged or destroyed. As a result of mechanical comminution, the surface of the encapsulated particles exposes the flavorings. An increased loading with surface flavorings is disadvantageously connected with this. In addition, an undesirably high fine dust content is formed.

The exposed flavoring on the surface of the particles forms an oily layer on the surface of the particle. This oily layer on the surface causes a marked impairment in the shelf life of the particles.

Therefore, a method to produce flavorings encapsulated in carbohydrates is sought. There is a need for a solvent–free continuous procedure, which involves the integration of the individual steps shaping, cooling, comminution, and dedusting. The particles produced by this sought method must be free from an oily layer from the flavoring used and have a high glass transition temperature. It is desirable for the resulting encapsulated flavorings to have a long shelf life and a high glass transition temperature.

SUMMARY OF THE INVENTION

A process is now provided by the present invention for producing carbohydrate-encapsulated flavorings, produced by emulsifying the flavoring into a carbohydrate melt and producing particles from the resultant melt, which is characterized in that the particles are treated with an inert gas.

The present invention also relates to carbohydrate-encapsulated flavoring particles, produced by emulsifying the flavoring into a carbohydrate melt, characterized in that the particles are treated with an inert gas.

The novel flavoring particles of the present invention are virtually free at the surface from an oily layer from the flavoring used and have a high glass transition temperature.

DETAILED DESCRIPTION OF THE INVENTION

A process is now provided by the present invention for producing carbohydrate-encapsulated flavorings, produced by emulsifying the flavoring into a carbohydrate melt and producing particles from the resultant melt, which is characterized in that the particles are treated with an inert gas.

The process of the present invention can be carried out either batchwise or continuously.

Inert gases for the process of the present invention are nitrogen, noble gases such as helium and argon, and air. The preferred inert gas is air.

The process of the present invention is generally carried out in the temperature range from 10 to 35°C, preferably 20 to 25°C.

The particles are treated with the gas stream by passing the inert gas through a convective dryer at a gas velocity of 0.2 to 4 m/s, preferably 0.5 to 2 m/s.

Convective dryers, for example fluidized-bed apparatuses, are used to remove the surface flavorings the carbohydrate-encapsulated flavoring particles.

The present invention also relates to carbohydrate-encapsulated flavoring particles, produced by emulsifying the flavoring into a carbohydrate melt, characterized in that the particles are treated with an inert gas.

The novel flavoring particles of the present invention are virtually free at the surface from an oily layer from the flavoring used and have a high glass transition temperature. The flavoring is located virtually exclusively in the interior of the particles.

Carbohydrates for the flavorings encapsulated according to the present invention include, but are not limited to hydrolyzed starches, mono- and/or disaccharides, such as maltose.

The flavoring particles have a cylindrical or spherical geometry and a narrow particle size profile. They have a diameter of 0.3 to 12 mm, preferably 0.5 to 1.0 mm, and a length of 0.3 to 10 mm, preferably 0.5 to 1 mm.

The particles of the present invention have a flavoring content of 1 to 25% by weight, preferably 3 to 10% by weight based on the entire weight of the particles.

The particles of the present invention have a glass transition temperature in the range from 45 to 75°C, preferably 50 to 60°C (DSC method, heating rate 20 K/min).

The flavoring particles of the present invention may comprise further substances, for example emulsifiers, colorants and other fillers.

The flavoring particles of the present invention can be used for flavoring food products, for example instant drink powders, tea, soup powders or sauce powders, confectionery products, chewing gum and pharmaceuticals and also consumer items.

The flavoring particles of the present invention can also be used for flavoring consumer items, for example, oral care products (toothpaste, denture cleaning tablets), cosmetic products, soaps, hygiene products, household products.

The flavoring particles of the present invention can also be used for pharmaceuticals, for example, tablets, candies, instant products.

Those skilled in the art could substitute odorants for flavorings according to the invention.

The process of the invention to produce carbohydrate-encapsulated flavoring particles can be carried out in the following steps:

Melt production

The first steps in the production are:

1) Melt dry mixture: A dry carbohydrate mixture is prepared and melted by heating to 80 to 120°C, preferably to 90 to 100°C, in an extruder. Double-screw extruders having a plurality of temperature zones are preferred.

2) Emulsify flavoring into the dry mixture: The flavoring, which can additionally contain a suitable emulsifier, is added via a pump continuously into the front extruder region at a dosage of 1 to 25, preferably 3 to 10, % by weight, based on the dry mixture. The extruder dyes ensure the emulsification of the flavoring in the melt.

Integration of the individual steps shaping, cooling and comminution

To shape the strands downstream of the perforated plate, the melt must be cooled. Concentric impinging of the perforated plate with cold air cools the melt strands. During this, attention must be paid to uniform heating / cooling of the perforated plate. The strands are comminuted while they are still in the solidification phase by dye-phase pelletizing. A gas-tight design of dye-phase pelletizer having rotary blades, for example, comminutes the strands into pellets. Variable rotary speed control of the dye-phase pelletizer adjusts the particle length as a function of solid throughput. The resultant particles have a bulk density of approximately 0.5 kg/l.

Process for removing the surface flavoring

In this process step, the inventive treatment of the particles is performed using an inert gas.

Contacting is effected in convective dryers by blowing, vortexing or mixing. In a fluidized-bed apparatus, a gas throughput is required which is equivalent to a superficial velocity of 0.2 to 4 m/s, preferably 0.5 to 2 m/s, and a flowthrough time in the range from 5 to 120 min, preferably from 20 to 40 min, at a filling level of 0.01 to 0.5 m, preferably 0.05 to 0.2 m.

Integrated dedusting

When the particles are contacted with inert gas, adhering or newly formed fine dust is entrained by the gas stream. The dust-laden exhaust gas can be conducted via a suitable dedusting system, so that downstream screening of the particles is unnecessary.

A screening product discharge such as a zigzag screen or ascending tube screen can be connected downstream of the convective dryer.

By means of the process of the present invention, encapsulated flavoring particles are obtained having a low surface flavoring loading by the flavoring and a high glass transition temperature.

The invention is explained in greater detail by means of the following examples.

Example 1 Production of lemon flavoring particles

Lemon flavoring is incorporated at 5% into a melt of various maltodextrins, disaccharides and an emulsifier in the extruder. Via a 0.5 mm perforated plate, strands are formed which are comminuted by means of dye-phase pelletizing to a length of 0.5 to 1 mm. 1 kg of the particles is then contacted for 60 minutes with air in a fluidized-bed apparatus operated batchwise. To fluidize the bed contents, air is blown in at a superficial velocity of 1.25 m/s. The inlet temperature of the fluidizing gas is 25°C. The temperature of the exhaust gas is 25°C. Dust contents are then removed via a 0.5 mm screen.

Example 2 Instant drink Powder

An instant drink-powder mixture is formulated consisting of 90% by weight of sucrose, 8% by weight of citric acid, 1% by weight of other ingredients (calcium phosphate, ascorbic acid, modified cellulose, dye) and 1% by weight of yellow-colored lemon flavoring particles (diameter 0.4-0.6 mm) which are produced according to the procedure described. The mixture exhibits a particularly long shelf life with respect to flavoring. Because of the oxidation-sensitive lemon flavoring on the surface of the flavoring particles, the production of off-notes (caused by oxidation) is very greatly minimized.

Example 3 Bagged tea

Flavoring of black tea in teabags with 3% by weight of strawberry flavoring particles (diameter 1 mm, length 1–2 mm). During storage of the tea, the flavoring remains encapsulated in the granular matrix, and is not released until the infusion by dissolving the particle matrix in hot water.

Example 4 Chewing gum

Chewing gum mass is admixed with blue-colored peppermint flavoring particles (diameter 0.6 mm, length 0.4 mm) which were produced by the process described by the present invention. The particles generate a special optical effect. The flavoring is released mechanically on chewing.

Example 5 Comparison

In the comparison below (Table 1), the flavoring particles of the invention are compared before and after the contact with air. It is shown that solvent-free decrease of the surface flavoring is possible using the process of the invention.

To determine the amount of flavoring on the surface of the resultant particles, the particles are washed with pentane/ether (particles are not dissolved in this process) and the wash liquid is analyzed for flavoring content with a gas chromatograph. The results of this experiment are reported in ppm based on initial weight of granules.

Table 1:

	Before air treatment	After air treatment (fluidized bed 40 min, 1.25 m/s, 25°C) (according to the invention)
Lemon granules (diameter 1 mm)	197 ppm	9 ppm
Strawberry granules (diameter 1 mm)	943 ppm	4 ppm

Although the invention has been described in detail in the foregoing for the purpose of illustration, it is to be understood that such detail is solely for that purpose and that

variations can be made therein by those skilled in the art without departing from the spirit and scope of the invention except as it may be limited by the claims.

WHAT IS CLAIMED IS:

1. A process for producing encapsulated flavorings, comprising the steps of emulsifying the flavoring into a carbohydrate melt, wherein said carbohydrate melt is prepared from a carbohydrate mixture melted by heating to 80 to 120°C, to form a resultant melt and producing particles from the resultant melt, wherein the particles are treated with an inert gas.

2. A process according to Claim 1, wherein said inert gas is air.

3. A process according to Claim 1, wherein said particles are treated with an inert gas in the temperature range from 10 to 35°C.

4. A process according to Claim 1, wherein said particles are treated in a gas stream at a gas velocity of 0.2 to 4 m/s.

5. Carbohydrate-encapsulated flavoring particles, produced by emulsifying the flavoring into a carbohydrate melt, wherein said carbohydrate melt is prepared from a carbohydrate mixture melted by heating to 80 to 120°C, to form a resultant melt and producing particles from the resultant melt, wherein the particles are treated with an inert gas.

6. Carbohydrate-encapsulated flavoring particles according to Claim 5, wherein said particles comprise a glass transition temperature of 45 to 75°C.

7. Carbohydrate-encapsulated flavoring particles according to Claim 5, wherein said particles comprise a diameter in the range from 0.3 mm to 12 mm and a length of 0.3 to 10 mm.

8. Carbohydrate-encapsulated flavoring particles according to Claim 5, wherein said particles comprise a flavoring content of 1 to 25% by weight based on the weight of the particles.

9. Carbohydrate-encapsulated flavoring particles according to Claim 5, wherein said carbohydrate mixture is selected from the group consisting of hydrolyzed starch, mono- and/or disaccharides.

10. Carbohydrate-encapsulated flavoring particles according to Claim 5, wherein said flavorings is selected from the group consisting of flavorings, natural extracts, nutraceuticals and/or food additives.

11. Foods comprising carbohydrate-encapsulated flavorings wherein said carbohydrate-encapsulated flavoring particles are produced by emulsifying the flavoring into a carbohydrate melt, wherein said carbohydrate melt is prepared from a carbohydrate mixture melted by heating to 80 to 120°C, to form a resultant melt and producing particles from the resultant melt, wherein the particles are treated with an inert gas.

12. Consumer articles comprising carbohydrate-encapsulated flavorings wherein said carbohydrate-encapsulated flavoring particles are produced by emulsifying the flavoring into a carbohydrate melt, wherein said carbohydrate melt is prepared from a carbohydrate mixture melted by heating to 80 to 120°C, to form a resultant melt and producing particles from the resultant melt, wherein the particles are treated with an inert gas.

13. Pharmaceuticals comprising carbohydrate-encapsulated flavorings wherein said carbohydrate-encapsulated flavoring particles are produced by emulsifying the flavoring into a carbohydrate melt, wherein said carbohydrate melt is prepared from a carbohydrate mixture melted by heating to 80 to 120°C, to

form a resultant melt and producing particles from the resultant melt, wherein the particles are treated with an inert gas.

ENCAPSULATED FLAVORINGS

ABSTRACT OF THE DISCLOSURE

A method for producing carbohydrate-encapsulated flavorings whose surface has been treated with an inert gas, as well as carbo-hydrate-encapsulated flavorings and the use thereof in food products, consumer articles and pharmaceuticals.

COMBINED DECLARATION AND POWER OF ATTORNEY	ATTORNEY DOCKET NO

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

Encapsulated Flavorings

the specification of which is attached hereto,

or was filed on _____ as

Application Serial No. _____

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s), the priority(ies) of which is/are to be claimed:

19954528.6	Germany	November 12, 1999
(Number)	(Country)	(Month/Day/Year Filed)

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose the material information as defined in Title 37, Code of Federal Regulations, §1.56 which occured between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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ARON PREIS, Patent Office Registration Number 29,426
LYNDANNE M. WHALEN, Patent Office Registration Number 29,457
THOMAS W. ROY, Patent Office Registration Number 29,582
RICHARD E. L. HENDERSON, Patent Office Registration Number 31,619
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NOLAND J. CHEUNG, Patent Office Registration Number 39,138
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